IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:
Murray Orpin

Confirmation No.: 4448

Application No.: 10/715,948

Group Art Unit: 1711

Filing Date: November 18, 2003

Examiner: Zemel, Irina Sopjla

For: A Syntactic Phenolic Foam Composition

DATE OF DEPOSIT:

I HEREBY CERTIFY THAT THIS PAPER IS BEING DEPOSITED WITH THE UNITED STATES POSTAL SERVICE AS FIRST CLASS MAIL, POSTAGE PREPAID, ON THE DATE INDICATED ABOVE AND IS ADDRESSED TO THE COMMISSIONER FOR PATENTS, P.O. BOX 1450, ALEXANDRIA, VA 22313-1450.

DECLARATION UNDER 37 C.F.R. § 1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Murray Orpin, Ph.D., declare as follows:

1. I am named as the inventor in the above-identified application.

2. I have been working in the field of phenolic resin technology for 16 years and am widely acknowledged to be a global authority in the area of reactive phenolic resins. My CV is attached hereto as Exhibit A.

3. I believe that pre-mixes and syntactic phenolic foam compositions, as well as processes for their preparation, as claimed in the above-identified application, require incorporation of highly reactive phenolic resole resins that may rightfully be described as unique since there is no evidence in any literature that they have been made or used at any time in past history.

4. The pre-mixes and syntactic phenolic foam compositions each require the incorporation of the highly reactive phenolic resole resins prepared at a reaction temperature that is under 65°C. Specifically, the process now requires that the resin is obtained by reacting a substituted or unsubstituted phenol and an aldehyde in the presence of an alkaline catalyst at a temperature of no more than 65°C. In fact, a reaction temperature of 58-62°C is preferred, whilst a temperature of about 60°C is most preferred. The required reaction temperature of under 65°C and the preferred reaction temperature of 58-62°C, although they would appear to be only slightly under that disclosed by Garrett, are critical for maximum methylolation of the aromatic phenolic ring by condensation with formaldehyde. Any reaction temperature above 63-64°C would lead to longer chain phenolic molecules, less available methylol groups and hence reduced reactivity to mild acid catalysts.

5. I declare that the final neutralisation pH of the desired highly reactive phenolic resole resin is between 5.5-6.6. Garrett merely cites a neutralisation pH of 'about 7'. By using the term "about", Garrett did not see the neutralisation pH as being important. Moreover, Garrett did not see the resin's reactivity as being important to his disclosure at all. It is also clear from column 2, lines 10-13 of Garrett that the resoles that Garrett described were not capable of tolerating high amounts of water whereas the resoles of the present application can cure with up to 10 times their weight of water present and are required to, in order to produce some of the materials cited in the Examples of the present application.

6. Under my direction and control, more data have now been generated to support the importance of the critical neutralisation pH range of 5.5-6.6, showing the effect on reactivity. Lab studies, under my direction and control, have shown that a Reaction/Gel time of 4-10 minutes is required for acceptable reactivity according to the test definition already provided. Data from the quality assurance (QA) testing of recent production batches, under my direction and control, now support the teaching that, above a pH of 6.6, the reactivity of the resin is rapidly lost (see below).

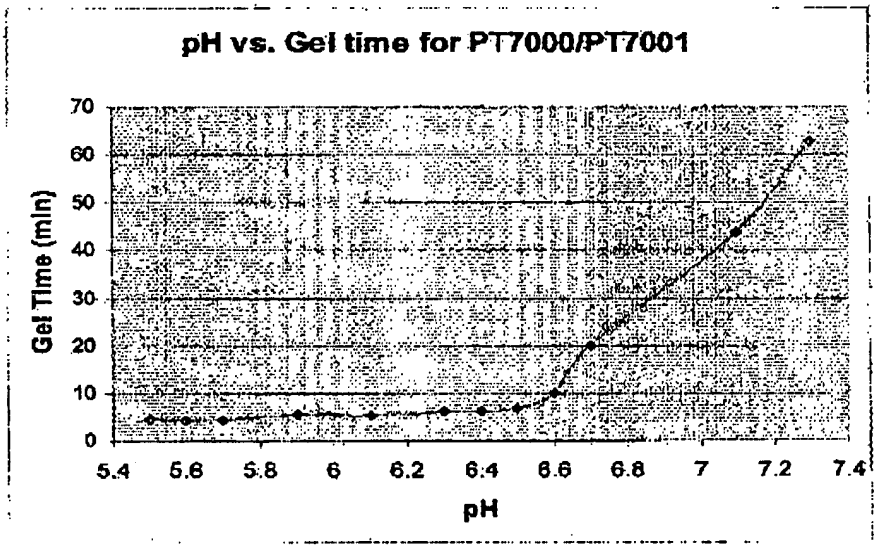
7. The Final Office Action suggests that pH measurements have been made to an accuracy of +/-0.5, referring to submissions made on behalf of Applicant in the response made to the first Office Action. I declare that this statement was in fact made in error since use of the '+/- 0.5' range was taken from text submitted by me which stated that the final neutralisation pH of the highly reactive phenolic resole resin should be pH 6 +/- 0.5. By this I meant that the most ideal neutralisation pH was between 5.5 and 6.5. This is roughly equivalent to the disclosed range of 5.5 to 6.6. I did not mean that my pH measurements were done with a precision of +/-0.5. The Examiner will appreciate that pH measurements may typically be made with far greater accuracy than +/- 0.5. I declare that the pH measurements provided below and those submitted in response to the First Office Action were measured to an accuracy of +/- 0.02.

8. The phenolic resole resin in question was prepared, under my direction and control, using the general teaching at pages 10 and 11 of the US application as filed. Specifically, 100 parts by weight of phenol was reacted with 116.5 parts by weight of 44% formaldehyde in the presence of 9.09 parts by weight of 25% sodium hydroxide solution. The mixture is then stirred and heated to a temperature of 60°C for a period of between 4 and 6 hours, until an intermediate viscosity of 13.5-14.5 centiStokes is reached at a temperature of 25°C. The mixture is then neutralised with *p*-toluenesulphonic acid to a desired pH (see below) (at an accuracy of +/- 0.02). Most of the process and reaction water is then distilled off under vacuum to a water level of 4% (+/- 1%). The thus obtained resole resin has a typical viscosity of 2000 – 3000 mPas at 25°C.

The reactivity test is the time taken (in minutes) for the catalysed resin to initiate exotherm, as evidenced by boiling and the onset of hardening. The reactivity test is carried out in the following manner. The resin is stabilised at 25°C. 50g of the resin is taken in a 200ml waxed paper cup. 0.5g of a catalyst comprising 85% xylene sulphonic acid/ 15% *o*-phosphoric acid (sold by Borden Chemicals as Phencat 15) is added with intermediate mixing and a stop-clock is started. Mixing is continued for 20 seconds, after which the cup is left in a well-ventilated and temperature-controlled area. The stop-clock is stopped at the exotherm onset point and the time is recorded.

The additional data in relation to the criticality of the pH range are presented below both in tabular and graphical form. Over 1.1pH units (between 5.5 and 6.6), the gel time doubles to 10 minutes at pH6.6. The gel time doubles, over 0.1pH units, between pH 6.6 and pH 6.7. These data support a pH neutralisation range of 5.5-6.6 as now recited in the claims. These data demonstrate that controlling the reaction temperature to no more than 65°C and controlling the neutralisation pH to within 5.5 to 6.6 unexpectedly yields a highly reactive phenolic resin, suitable for pre-mixes and syntactic phenolic foam compositions.

pH	Reaction/Gel time (min)
5.5	4.75
5.6	4.3
5.7	4.3
5.9	5.6
6.1	5.15
6.3	6.33
6.4	6.24
6.5	7
6.6	10
6.7	20
7.1	44
7.3	63



9. Garrett's Example 1 discloses that the resole resin is prepared by reacting phenol and an aldehyde (formaldehyde) in the presence of an alkaline catalyst (sodium hydroxide) at 65°C, followed by neutralisation to a pH of about 7.0 (see column 6, lines 21-28). There is no disclosure or suggestion that the reactivity of the resole resin can be altered by altering the neutralisation pH below the "about 7.0" disclosed in Garrett. Garrett describes the use of alkaline glass additives as a scavenger for the strong acid catalysts required by the type of resole given as an example therein but the present invention is able to produce resole-based foams that are inherently pH neutral without the use of any scavenger. This is just one of many beneficial qualities of the products that may be produced with the highly reactive resole resins. Other unique characteristics are:

- The ability to fully cure at ambient temperature.
- The ability to achieve full cure without the need of a post-cure cycle at elevated temperature.
- The ability to fully cure in the presence of up to 10 times their own weight in water.
- Very low water content at a readily processable viscosity.
- Very high exotherm temperatures with mild catalysts.
- Extremely rapid cure where required.

10. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the above-referenced application or any patent issuing thereon.

14th November, 2005 Murray Orpin

Date